# USAXS Characterization of Materials

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*Ultra-small-angle X-ray scattering (USAXS) experiments supply data in the usually inaccessible Q range (where $Q = (4\bullet /\lambda)\sin\theta$, $\lambda$ is the x-ray wavelength and $\theta$ is one-half the angle of scatter) down to $1.5 \times 10^{-4}$ Å$^{-1}$. In that role, USAXS fills the gap between visible light scattering and pinhole small angle cameras. As one of the few small-angle instruments in the world for which a primary absolute calibration is available, the results from the NIST USAXS facility are quantitative rather than qualitative, which is of great importance to serious materials science research.*

The ultra-small-angle X-ray scattering (USAXS) instrument on UNICAT sector 33 at the Advanced Photon Source provides data in the usually inaccessible scattering vector, Q range (where $Q = (4\bullet /\lambda)\sin\theta$, $\lambda$ is the x-ray wavelength and $\theta$ is one-half the angle of scatter) down to $1.5 \times 10^{-4}$ Å$^{-1}$. This unusual instrument covers nearly 4 decades in Q, and 12 decades in linear dynamic range in X-ray intensity. In the past year, its function has been extended to the measurement of anisotropic USAXS and USAXS imaging.

To remove the intrinsic slit-smearing of the USAXS instrument, transverse crystal reflections orthogonal to the plane of the collimator and analyzer crystal reflections are introduced. The data in a single scan are associated with one azimuthal direction in the plane of the sample. Thus, the sample is measured repeatedly for different azimuthal orientations, where the resolution of the azimuthal rotation increments can be made almost vanishingly fine. Indeed, the anisotropic resolution of the new instrument can surpass that of conventional pinhole instruments with a 2D detector.

Studies of textured $Fe_2TiO_5$ have been carried out with reference to numerical model predictions based on the OOF program of NIST's CTCMS. Fig. 1 shows the anisotropic scattering associated with cracks perpendicular to a strong fiber- texture axis (strong maximum along the direction, **B**) and also a 2D random distribution of microcracks with planes parallel to the direction, **B** (maximum around equator perpendicular to **B**).

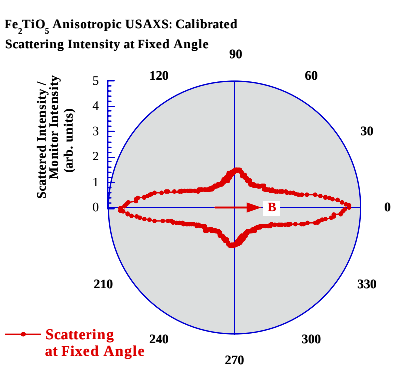


Fig. 1: USAXS of textured iron titanate.

USAXS imaging is a completely new class of X-ray imaging technique that is remarkably sensitive to the microstructures within a scattering volume. In USAXS imaging, the analyzer is rotated to a specific angle and the selected X-rays are used to form an image of the sample. Since the *only* X-rays that contribute to the image are those produced by small-angle scattering, the image is a direct map of where the USAXS is originating from within the sample. Information on the sizes and shapes of the scattering objects can be obtained by comparing images produced at different scattering angles. Such information can be determined even when the scattering objects are smaller than the spatial resolution of the imaging process! The image contrast doesn't change during sample rotations about the scattering vector (vertical axis in the lab) and combining images from two such rotations has produced stereo USAXS images. In principle, a full 3D tomographic reconstruction should also be feasible. USAXS imaging should prove useful both as an independent imaging technique and as an important adjunct to USAXS generally. It is likely to find application to a broad range of materials problems in metal, ceramic, polymer and biological systems.

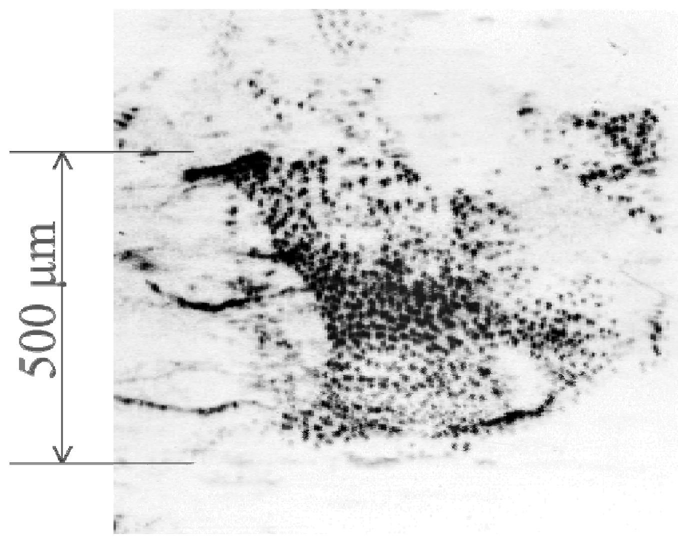


Fig 2: USAXS image of creep cavities in copper.

## Contributors and Collaborators

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